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(54) LUBRICATING AGENT FOR MOLD AT ELEVATED TEMPERATURE, IRON-BASED POWDER COMPOSITION FOR ELEVATED TEMPERATURE COMPACTION WITH LUBRICATED MOLD AND HIGH DENSITY FORMED PRODUCT FROM IRON-BASED POWDER COMPOSITION, AND METHOD FOR PRODUCING HIGH DENSITY IRON-BASED SINTERED COMPACT

(57) A process for producing a high-density iron-based green compact is provided which can form a green compact with a high density. Also provided is a process for producing a sintered compact from the green compact. A specified combination lubricant is applied to the surface of a die for pressure compaction by electrical charging, which lubricant is composed of a lubricant having a higher melting point than a preset compaction temperature, and a lubricant having a lower melting point than the compaction temperature. A heated iron-based powder mixture is filled into the die, followed by pressure compaction, whereby a green compact is formed. The green compact can be sintered to provide a sintered compact. The powder mixture comprises an iron-based powder, a powder compaction lubricant and a graphite powder, wherein the powder compaction lubricant comprises a lubricant having a lower melting point than the compaction temperature and in a content from 10 to 75% by mass, and a lubricant having a higher melting point than the compaction temperature and as the balance, and the content of the graphite powder is less than 0.5% by mass based on the total amount of the iron-based powder mixture.

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Description

BACKGROUND OF THE INVENTION

1. Field of the Invention

[0001] This invention relates to processes for the production of iron-based powder green compacts and iron-based sintered compacts for powder metallurgy. More particularly, the invention relates to improvements in lubricants for use in producing a high-density, green compact made from iron-based powder by warm compaction.

2. Description of the Related Art

[0002] In general, a powdered iron-based green compact for powder metallurgy is produced by filling an iron-based powder mixture into a die. The powder mixture is generally derived by mixing an iron-based powder with alloying powders such as copper powder, graphite powder and the like and further with lubricants such as zinc stearate, lead stearate and the like, and then by subjecting the iron-based powder mixture to. The resultant green compact usually has a density in the range from 6.6 to 7.1 Mg/m^3.

[0003] Such a green compact is further sintered to obtain a sintered compact which, where desired, is sized or cut into a powder metallurgical product. Where great strength is required, carburizing heat treatment or brightening heat treatment is in some instances performed after completion of the sintering.

[0004] The above powder metallurgy permits components parts of complicated shapes to be formed with high dimensional accuracy and in near net structure, significantly saving the cost of cutting work as contrasted to conventional production methods.

[0005] With regard to powder metallurgical iron products, a keen demand has recently been made for more higher dimensional accuracy to omit cutting work and to save production cost, and also for more greater strength to make components parts small in size and light in weight.

[0006] In order to give greater strength to a powder metallurgical product (a sintered compact), it is beneficial to form high-density sintered compacts from an iron-based green compact which has been produced to have a high density. As the density of a sintered compact increases, the number of voids in the compact decreases so that the component part is obtainable with improved mechanical properties such as tensile strength, impact value, fatigue strength and the like.

[0007] As warm compaction techniques evolved to form a high-density iron-based green compact, there have been proposed a double molding-double sintering method in which an iron-based powder mixture is pressed and sintered in the usual manner, followed by repeated pressing and sintering, and a sinter forging method in which single pressing and single sintering are performed, followed by hot forging.

[0008] Moreover, warm compaction techniques are known in which metal powder is compacteds with heat as disclosed for instance in Japanese Unexamined Patent Application Publication No. 2-156002, Japanese Examined Patent Application Publication No. 7-103404, U.S. Patent No. 5,256,185 and U.S. Patent No. 5,368,630. Such a warm compaction techniques are designed to melt and disperse a lubricant partly or wholly between the metallic particles, thereby reducing the frictional resistance between the metallic particles and the frictional resistance between the green compact and an associated die, so that improved compressibility is attained. The compaction technique noted here is thought to be most advantageous in view of possible cost savings amongst the methods previously mentioned for the production of high-density iron-based green compacts. A green compact of about 7.30 mg/m^3 in density can be obtained by the above compaction technique when an iron-based powder mixture is compacted at a pressure of 7 t/cm^2 and at a temperature of 150 C, which powder mixture is derived by mixing a partially alloyed iron powder of a Fe-4Ni- 0.5Mo-1.5Cu with 0.5% by mass of graphite and 0.6% by mass of lubricant.

[0009] However, according to the warm compaction techniques of the above-cited publications, i.e., Japanese Unexamined Patent Application Publication No. 2-156002, Japanese Examined Patent Application Publication No. 7-103404, U.S. Patent No. 5,256,185 and U.S. Patent No. 5,368,630, the problem arises that an iron-based powder mixture is less fluid and hence less productive, the resultant green compact is irregular in respect of densities, and the resultant sintered compact is unfavorably variable in respect of physical properties. Another drawback is that a high force must be applied to draw the green compact from the corresponding mold with consequent marred surface of the product and shortened lifetime of the die.

[0010] In these warm compaction techniques, a lubricant is also contained in an iron-based powder mixture so as to reduce the resistance between the metallic particles and the resistance between the green compact and the associated mold, thereby providing improved conpressibility. During warm compaction, the lubricant is partly or wholly melted and then pushed to locate adjacent to the surface of the green compact. Upon subsequent sintering, the lubricant gets thermally decomposed or volatilized and hence escapes from the green compact, leaving coarse voids near to the

surface of the sintered compact. This poses the problem that the sintered compact results in insufficient mechanical strength.

[0011] To cope with this problem, Japanese Unexamined Patent Application Publication No. 8-100203 discloses that when room temperature compaction or warm compaction is effected, the content of a lubricant to be incorporated in an iron-based powder mixture is decreased by coating the surface of a die with an electrical charged lubricant powder such that a high-density green compact is produced. In this technique, however, the coating lubricant is susceptible to morphological changes at around its melting point since it is of a single nature so that the lubricating action is largely variable. This has the drawback that the compaction temperature range depends restrictedly upon the melting point of the coating lubricant. Also defectively, even if the content of the lubricant in the powder mixture can be decreased with the coating lubricant applied on to the mold surface, the content of the former lubricant may be too low which is dependent upon the lubricant components to be incorporated in the powder mixture. In this instance, the former lubricant does not exhibit lubrication, failing to enhance the density of a pressurized powder.

[0012] From the viewpoints of great strength and cost saving of automotive parts, there has been a need for the development of a process capable of producing an iron-based green compact with a higher density but by single compaction.

SUMMARY OF THE INVENTION

[0013] In order to eliminate the foregoing problems of the conventional art, a first object of the present invention is to provide a process for producing a high-density iron-based green compact which permits a high-density green compact to be formed with a density of 7.4 Mg/m^3 or above and by single pressing when warm pressure compaction is effected as to an iron-based powder mixture derived by mixing a partially alloyed iron powder of, for example, a Fe-4Ni-0.5Mo-1.5Cu composition, with 0.5% by mass of a graphite powder.

[0014] A second object of the invention is to provide a process for producing a high-density iron-based sintered compact which permits a high-density sintered compact to be formed by sintering such an iron-based green compact.

[0015] To achieve the above objects by utilizing a warm compaction technique and a die lubrication compaction technique, the present inventors have conducted intensive researches on various lubricants for mold lubrication and various formulations of iron-based powder mixtures containing lubricants. It has now been found that the force for drawing an iron-based green compact from the corresponding mold can be effectively lessened by the use of a certain specific combination lubricant as a lubricant for mold lubrication. This combination lubricant is composed in a suitable ratio of a lubricant having a lower melting point than a preset compaction temperature and a lubricant having a higher melting point than the compaction temperature and can be applied to the surface of a preheated die by electrical charging.

[0016] The present invention has been made on the bass of the aforesaid finding and further supporting studies.

[0017] More specifically, according to a first aspect of the present invention, there is provided a lubricant for warm mold lubrication, comprising a mixture of a lubricant having a higher melting point than a preset compaction temperature, and a lubricant having a lower melting point than the compaction temperature, the lubricant for mold lubrication being applicable to the surface of a preheated die by means of electrical charging when a powdered material is compacted in the mold by pressure compaction.

[0018] According to a this invention, there is provided a die lubricant for warm compaction with die, comprising a lubricant having a higher melting point than a preset compaction temperature and in a content from 0.5 to 80% by mass, and a lubricant having a lower melting point than the compaction temperature and as the balance, the lubricant being applicable to a surface of a preheated die by means of electrical charging when a powdered material is compacted in the mold by pressure compaction.

[0019] In this aspect, the higher-melting lubricant is at least one selected from the group consisting of metallic soap, thermoplastic resin, thermoplastic elastomer, and an organic or inorganic lubricant having a lamellar crystal structure.

[0020] In this aspect, the lower-melting lubricant is at least one selected from the group consisting of metallic soap, amide wax, polyethylene, and an eutectic mixture of at least two members thereof.

[0021] According to a second aspect of the invention, there is provided an iron-based powder mixture for warm compaction with die lubrication, comprising an iron-based powder and a powder compaction lubricant, wherein the powder compaction lubricant comprises a lubricant having a lower melting point than a preset compaction temperature and in a content from 10 to 75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance.

[0022] According to this aspect of the invention, there is provided an iron-based powder mixture for warm compaction with die lubrication, comprising an iron-based powder, a powder compaction lubricant and a graphite powder, wherein the powder compaction lubricant comprises a lubricant having a lower melting point than a preset compaction temperature and in a content from 10 to 75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance, and the content of the

graphite powder is less than 0.5% by mass based on the total amount of the iron-based powder mixture.

[0023] In the second invention, the content of the powder compaction lubricant is in the range from 0.05 to 0.40% by mass.

[0024] According to the third invention, there is provided a process for the production of a high-density iron-based green compact, comprising the steps of: preheating a die at a selected temperature; applying a die lubricant for warm compaction with die to the surface of the mold by electrical charging; filling a heated iron-based powder mixture in the mold; and then subjecting the mixture to pressure compaction at a preset compaction temperature, wherein the die lubricant for warm compaction with die lubrication comprises a lubricant having a higher melting point than the compaction temperature and in a content from 0.5 to 80% by mass, and a lubricant having a lower melting point than the compaction temperature and as the balance; and the iron-based powder mixture comprises an iron-based powder and a powder compaction lubricant, the powder compaction lubricant comprising a lubricant having a lower melting point than the compaction temperature and in a content from 10 to 75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance.

[0025] In this third invention, the graphite powder can be also added in a content less than 0.5% by mass based on the total amount of the iron-based powder mixture.

[0026] In the third invention, the higher-melting lubricant is at least one selected from the group consisting of metallic soap, thermoplastic resin, thermoplastic elastomer, and an organic or inorganic lubricant having a layer crystal structure.

[0027] The lower-melting lubricant is at least one selected from the group consisting of metallic soap, amide wax, polyethylene, and an eutectic mixture of at least two members thereof.

[0028] The lubricant for in the powder mixture is preferably added in a amount from 0.05 to 0.40% by mass.

[0029] The present invention can also provide a high-density sintered compact produced by single pressing.

[0030] In a fourth embodyment of the invention, there is provided a process for the production of a high-density iron-based sintered compact, comprising the step of further sintering the high-density iron-based green compact produced by the process according to any one of the fifth and sixth aspects, thereby forming a sintered compact.

[0031] The above and other objects, features and advantages of the present invention will become manifest upon reading of the following detailed description.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0032] In the practice of the present invention, a heated iron-based powder mixture is filled in a die and then molded by pressure compaction at a preset compaction temperature, whereby an iron-green compact is obtained.

[0033] In the invention, a die to be used is preheated at a suitable temperature. The preheating temperature is not particularly restricted so long as an iron-based powder mixture can be maintained at a preset compaction temperature. The preheating temperature is set to be preferably higher than the compaction temperature by 20 to 60 C.

[0034] An electrically charged lubricant for mold lubrication is introduced into a preheated die to apply the lubricant to the surface of the mold by electrical charging. Desirably, the lubricant (solid powder) for mold lubrication is placed in a die lubricating system (for example, Die Wall Lubricant System manufactured by Gasbarre Co.) where electrical charging is performed by means of contact charging between the solid lubricant particles and the inner wall of the system. The electrically charged lubricant is jetted into the mold and applied to the mold surface by electrical charging. The amount of the lubricant to be applied to the mold surface by electrical charging is set preferably in the range from 5 to 100 g/m^2. Amounts less than 5 g/m^2 result in insufficient lubricating action, calling for a high force to draw the resultant green compact from the mold. Amounts more than 100 g/m^2 cause the lubricant to remain on the product surface, making the product unsightly in appearance.

[0035] The die lubricant for warm compaction with die lubrication is used in electrically charged relation to the surface of a preheated die when a powdered material is compacted by pressure compaction. This lubricant is a mixture of a lubricant having a higher melting point than a preset compaction temperature and in a content from 0.5 to 80% by mass, and a lubricant having a lower melting point than the compaction temperature and as the balance. The preset compaction temperature used herein denotes a temperature as measured on the mold surface at the time pressure compaction is carried out.

[0036] The higher-melting lubricant is present in a sold state in the die lubricant for warm compaction with die lubrication at the time compaction is effected, and it behaves like a solid lubricant that acts as "a roller" within a die, consequently lessening the force for drawing a green compact from the mold. Moreover, such higher-melting lubricant has a role to prevent a completely or partially molten lubricant (a lower-melting lubricant to be described later) from getting migrated within the mold, decreasing the frictional resistance between the green compact and the mold surface so that the force for product drawing is prevented from being unfavorably increased.

[0037] If the content of the higher-melting lubricant is less than 0.5% by mass, the lower-melting lubricant becomes

relatively abundant. This causes a large amount of a molten lubricant that migrates within a die and does not distribute uniformly on the surface of the mold, increasing the frictional resistance between the green compact and the mold surface and hence failing to lessen the force for product drawing to a sufficient extent. Conversely, if the content of the higher-melting lubricant is more than 80% by mass, a lubricant not subject to melting in a die is too large in amount for uniform distribution on the die surface. This is responsible for diminished mold lubrication and hence for increased force for product drawing. Hence, the content of the higher-melting lubricant present in the die lubricant for warm compaction with die lubrication should be within the range from 0.5 to 80% by mass.

[0038] The lubricant for mold lubrication contains, in addition to the above-specified higher-melting lubricant, a lubricant having a lower melting point than the preset compaction temperature. This lower-melting lubricant melts completely or partially at the compaction temperature and presents a grease-like state on the surface of a die, exerting a beneficial effect on lessening the force for drawing a green compact from the mold.

[0039] The higher-melting lubricant is preferably at least one selected from the group consisting of metallic soap, thermoplastic resin, thermoplastic elastomer, and an organic or inorganic lubricant having a lamellar crystal structure. Suitable examples are chosen from the following lubricants depending upon the compaction temperature used.

[0040] As the metallic soap, zinc stearate, lithium stearate, lithium hydroxystearate or the like is preferred. As the thermoplastic resin, polystyrene, polyamide, fluorine resin or the like is preferred. As the thermoplastic elastomer, polystyrene elastomer, polyamide elastomer or the like is preferred. The inorganic lubricant of a lamellar crystal structure is graphite, MoS_2 or carbon fluoride, and finer particle sizes are more effective in lessening the force for product drawing. The organic lubricant of a lamellar crystal structure is melamine-cyanuric acid adduct (MCA) or N-alkyl aspartate- -alkyl ester.

[0041] Meanwhile, the lower-melting lubricant is desired to be a lubricant that melts completely or partially at the compaction temperature and tends to applied to the surface of a die at a low melting point by electrical charging. This lower-melting lubricant is preferably at least one selected from the group consisting of metallic soap, amide wax, polyethylene, and an eutectic mixture of at least two members thereof. Suitable examples are chosen from the following lubricants depending upon the compaction temperature used. As the metallic soap, zinc stearate or calcium stearate is preferred. As the amide wax, ethylene bis-stearoamide, monoamide stearate or the like is preferred. As the eutectic mixture, ethylene bis-stearoamide-polyethylene eutectic, ethylene bis-stearoamide-zinc stearate eutectic, ethylene bis-stearoamide-calcium stearate eutectic is preferred.

[0042] Subsequently, a heated iron-based powder mixture is charged into a die electrically charged with a lubricant for mold lubrication, followed by pressure compaction, whereby a green compact is obtained.

[0043] The iron-based powder mixture is heated preferably at from 70 to 200 C. Lower temperatures than 70 C result in an iron powder having increased yield stress and hence lead to a green compact having decreased density. Inversely, higher temperatures than 200 C show no appreciable rise in density, arousing a fear of an iron powder getting oxidized. Thus, the temperature at which the iron-based powder mixture is heated should be set within the range from 70 to 200 C.

[0044] The iron-based powder mixture is derived by mixing an iron-based powder with a lubricant (a powder compaction lubricant) or an alloying powder. The method of mixing the iron-based powder with the compaction lubricant or the alloying powder is not particularly restrictive, but any known method is suitably useful. In the case where the iron-based powder is mixed with the alloying powder, it is desired that after completion of primary mixing in which the iron-based powder and alloying powder are mixed with a part of the powder compaction lubricant, secondary mixing be performed in which the resultant mixture is stirred with heat at a higher temperature than the melting point of at least one of the aforesaid lubricant in order to melt the one lubricant, and the mixture having been melted is cooled with stirring to thereby apply the one lubricant to the surface of the iron-based powder mixture so that the alloying powder is bonded, followed by mixing of the balance of the powder compaction lubricant.

[0045] The iron-based powder according to the present invention is selected from among pure iron powders such as an atomized iron powder, a reduced iron powder or the like, a partially diffusively alloyed steel powder, a completely alloyed steel powder, and a mixed powder thereof.

[0046] The content of the powder compaction lubricant in the iron-based powder mixture is set preferably in the range from 0.05 to 0.40% by mass based on the total amount of the iron-based powder mixture. Contents less than 0.05% by mass make the resultant iron-based powder mixture less fluid and fail to apply the lubricant uniformly to the surface of a die, producing a green compact having decreased density. Conversely, contents more than 0.40% by mass suffer high voiding after sintering and give a sintered compact having decreased density.

[0047] The powder compaction lubricant contained in the iron-based powder mixture is a mixed lubricant obtained by mixing a lubricant having a lower melting point than the preset compaction temperature and a lubricant having a higher melting point than the compaction temperature. The content of the lower-melting lubricant in the powder compaction lubricant is in the range from 10 to 75% by mass, whereas the content of the higher-melting lubricant is in the range from 25 to 90% by mass as the balance. The lower-melting lubricant is effective in that it melts during pressure compaction, penetrates in between the iron-based particles by capillary action, disperses uniformly in the particles, reduces particle-to-particle contact resistance and facilitates reorientation of iron-based particles, thus accelerating

the enhancement of product density. If the content of the lower-melting lubricant is less than 10% by mass, such lubricant fails to disperse uniformly in the iron-based particles and suffers poor density of a green compact. If the content of the lower-melting lubricant is more than 75% by mass, a molten lubricant is squeezed toward the surface of a die as the density of a green compact is increased so that passages are provided on the product surface for the molten lubricant to escape out of the product. The passages cause many coarse voids on the product surface, giving insufficient strength to the resultant sintered compact.

[0048] The higher-melting lubricant contained in the iron-based powder mixture is present in a solid state at the time compaction is effected. This lubricant acts as "a roller" on the surface protrusions of iron-based particles where it repels a molten lubricant, promoting particle reorientation and enhancing product density.

[0049] The higher-melting lubricant contained in the powder compaction lubricant for the iron-based powder mixture is preferably at least one selected from the group consisting of metallic soap, thermoplastic resin, thermoplastic elastomer, and an organic or inorganic lubricant having a lamellar crystal structure. Suitable examples are chosen from the following lubricants depending upon the compaction temperature used.

[0050] As the metallic soap, zinc stearate, lithium stearate, lithium hydroxystearate or the like is preferred. As the thermoplastic resin, polystyrene, polyamide, fluorine resin or the like is preferred. As the thermoplastic elastomer, polyethylene elastomer, polyamide elastomer or the like is preferred. As the inorganic lubricant of a lamellar crystal structure, graphite, MoS_2 or carbon fluoride is preferred, and finer particle sizes are more effective for lessening the force for drawing a green compact from a die. As the organic lubricant of a lamellar crystal structure, melamine-cyanuric acid adduct (MCA) or N-alkyl aspartate- -alkyl ester is preferred.

[0051] The lower-melting lubricant contained in the powder compaction lubricant for the iron-based powder mixture is preferably at least one selected from the group consisting of metallic soap, amide wax, polyethylene, and an eutectic mixture of at least two members thereof. Suitable examples are chosen from the following lubricants depending upon the compaction temperature used.

[0052] As the metallic soap, zinc stearate, calcium stearate or the like is preferred. As the amide wax, ethylene bis-stearoamide, monoamide stearate or the like is preferred. As the eutectic mixture, ethylene bis-stearoamide-polyethylene eutectic, ethylene bis-stearoamide-zinc stearate eutectic, ethylene bis-stearoamide-calcium stearate eutectic or the like is preferred. Though dependent upon the compaction temperature used, some of these lower-melting lubricants may be utilized as higher-melting lubricants.

[0053] Graphite can be used as an alloying powder in the iron-based powder mixture. This graphite powder is effective to reinforce a sintered compact to be produced, but too high a content is liable to decrease product density largely. Hence, the content of graphite should be set to be less than 0.5% by mass based on the total amount of the iron-based powder mixture.

[0054] In the present invention, the high-density iron-based green compact formed by the above-specified production process can be further sintered, whereby a high-density iron-based sintered compact is obtained. Here, any conventional sintering method may be suitably used without limitation. Sinter hardening may also be used by which rapid cooling is effected after sintering to enhance product strength.

[0055] The present invention may be more fully understood with reference to the following examples.

Example 1

[0056] A partially alloyed steel powder of a Fe-4Ni-0.5Mo-1.5Cu composition derived by diffusively bonding Ni, Mo and Cu to a pure atomized iron powder was used as an iron-based powder. Iron-based powder mixtures were prepared by mixing this alloyed steel powder with 0.5% by mass of a graphite powder and various lubricants shown in Table 1. The mixing was effected with heat and by use of a high-speed mixer.

[0057] First, a die for compacting was preheated at each of the temperatures listed in Table 1. A die lubricant for warm compaction with die electrically charged by a die lubricating system (manufactured by Gasbarre Co.) was jetted into the die and applied to the mold die surface by means of electrical charging. The die lubricant for was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants shown in Table 2, and then by formulating both lubricants as shown in Table 1. The temperature measured on the mold surface was taken as a pressure compaction temperature.

[0058] Subsequently, the mold thus treated was filled with a heated iron-based powder mixture, followed by pressure compaction, whereby a rectangular green compact with a size of 10 x 10 x 55 mm was produced. The pressure loading was 686 MPa, and other pressure compaction conditions were as listed in Table 1. A powder compaction lubricant contained in the iron-based powder mixture was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants listed in Table 2, and then by formulating both lubricants as shown in Table 1.

[0059] As a conventional example, a similar rectangular green compact (Green compact No. 38) was formed in the same manner as in Example 1 except that a die was not coated with a die lubricant for warm compaction with die.

[0060] After completion of the compaction, the force was measured which was required for the green compact to be

drawn from the mold.

[0061] With regard to each green compact thus formed, the density was determined by Archimedes' principle. The principle noted here denotes a method by which the density of a test specimen, each green compact in this case, is determined by measuring the volume of the product after immersion in ethyl alcohol. Additionally, visual inspection was made of the appearance of the green compact to find faults such as marring, breakage and the like. The green compact was centrally cut, embedded in resin and then abraded, followed by examination of voiding in section on a light microscope.

[0062] The drawing force, density, appearance and sectional structure of the green compact are tabulated in Table 1.

[0063] All the green compacts representing this invention show as low a drawing force after compaction as 20 MPa or below and as high a density as 7.4 Mg/m^3 or above. Furthermore, these products are free of surface oxidation due to heating as well as faults such as marring, breakage and the like. The sectional structures are normal with the absence of coarse voids.

[0064] The comparative and conventional examples that fall outside the scope of the invention revealed a high drawing force exceeding 20 MPa, a low density of less than 7.35 Mg/m^3, or coarse voids near to the sectional surface of the green compact.

[0065] Advantageously, the present invention can form a high-density green compact which exhibits superior appearance and sectional structure and low drawing force.

Example 2

[0066] The following six different powders were used as iron-based powders; namely (1) a partially alloyed steel powder a of a Fe-4Ni-0.5Mo-1.5Cu composition derived by diffusively bonding Ni, Mo and Cu to a pure atomized iron powder, (2) a partially alloyed steel powder b of a Fe-2Ni-1Mo composition derived by diffusively bonding Ni and Mo to a pure atomized iron powder, (3) a prealloyed steel powder c of a Fe-3Cr-0.3Mo-0.3V composition derived by prealloying Cr, Mo and V, (4) a prealloyed steel powder d of a Fe-1Cr-0.3Mo-0.3V composition derived by prealloying Cr, Mo and V, (5) an atomized iron powder e, and (6) a reduced iron powder f. The atomized iron powder denotes an iron-based powder resulting from atomization of molten steel with high-pressure water, and the reduced iron powder denotes an iron-based powder resulting from reduction of iron oxide.

[0067] The partially alloyed steel powder a, partially alloyed steel powder b, prealloyed steel powder c, prealloyed steel powder d, atomized iron powder e and reduced iron powder f were each mixed with graphite in the contents shown in Table 3 and with the lubricants shown in Table 3, whereby iron-based powder mixtures were prepared. The mixing was effected with heat and by use of a high-speed mixer. In case of the atomized iron powder e and reduced iron powder f, 0.8% by mass of graphite and 2.0% by mass of a Cu powder were mixed. The content of graphite is by a mass ratio relative to the total amount of iron-based powder and graphite, or of iron-based powder, graphite and alloy powder.

[0068] First, a pressure compaction die was preheated at each of the temperatures listed in Table 3. A die lubricant for warm compaction with die electrically charged by a die lubricating system (manufactured by Gasbarre Co.) was jetted into the mold and applied to the mold surface by means of electrical charging. The die lubricant for warm compaction with die lubrication was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants shown in Table 2, and then by formulating both lubricants as shown in Table 3. The temperature measured on the mold surface was taken as a pressure compaction temperature.

[0069] Secondly, the mold thus treated was filled with a heated iron-based powder mixture, followed by pressure compaction, whereby a rectangular green compact with a size of 10 × 10 x 55 mm was produced. The pressure loading was 686 MPa, and other pressure compaction conditions were as listed in Table 3. A powder compaction lubricant contained in the iron-based powder mixture was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants listed in Table 2, and then by formulating both lubricants as shown in Table 3.

[0070] With regard to each iron-based green compact thus obtained, the density was determined by Archimedes' principle as in Example 1.

[0071] Subsequently, the iron-based green compact was sintered in a N_2-10%H_2 atmosphere and at 1,130 C for 20 minutes, whereby an iron-based sintered compact was formed. The density of the sintered compact was determined by Archimedes' principle. This product was then machined to obtain a sample in the shape of a small round rod dimensioned to be 5 mm in parallel plane diameter and 15 mm in length. The sample used to measure tensile strength.

[0072] Similar rectangular green compacts were formed in the same manner as in Example 2 except that a die was not coated with a die lubricant for warm compaction with die. Each green compact was further sintered as in Example 2 to form an iron-based sintered compact which was taken as a conventional example.

[0073] The test results are tabulated in Table 3.

[0074] The present invention provides high density and great tensile strength in contrast to the conventional examples (Sintered compacts Nos. 2 to 12).

Table 1-1

Green compact Nos.	Die lubricants: Lubricant of higher melting point than compacting temperature: Content* mass%	Die lubricants: Lubricant of higher melting point than compacting temperature: Kind (melting point)	Die lubricants: Lubricant of lower melting point than compacting temperature: Content* mass%	Die lubricants: Lubricant of lower melting point than compacting temperature: Kind (melting point)	Lubricants in powdered iron-based mixtures: Lubricant content** mass%	Lubricants in powdered iron-based mixtures: Lubricant of higher melting point than compacting temperature: Kind (melting point): Content by mass%***	Lubricants in powdered iron-based mixtures: Lubricant of higher melting point than compacting temperature: Content* mass%	Lubricants in powdered iron-based mixtures: Lubricant of lower melting point than compacting temperature: Kind (melting point): Content by mass%***	Lubricants in powdered iron-based mixtures: Lubricant of lower melting point than compacting temperature: Content* mass%	Compacting conditions: Die preheating temperature °C	Compacting conditions: Heating temperature for powdered iron-based mixture °C	Compacting conditions: Compacting temperature °C	Green compacts: Ejection force MPa	Green compacts: Density Mg/m³	Green compacts: Appearance	Green compacts: Sectional structure	Remarks
1	5	A3(230°C)	95	A1(148 to 155°C)	0.050	A3(230°C):0.025	50	A1(148 to 155°C): 0.025	50	210	150	160	17	7.40	good	good	Invention
2	5	A4(216°C)	95	A2(127°C)	0.200	A3(230°C):0.150	75	A1(148 to 155°C): 0.050	25	210	150	160	18	7.41	good	good	Invention
3	5	E1(240°C)	95	B1(146°C)	0.350	A3(230°C):0.200	57	C1(147°C):0.150	43	220	155	170	18	7.41	good	good	Invention
4	10	E2(260°C)	90	A2(127°C)	0.150	A4(216°C):0.050	33	A2(127°C):0.100	67	180	120	130	20	7.39	good	good	Invention
5	10	E3(316°C)	90	D1(<147°C)	0.050	A4(216°C):0.025	50	A1(148 to 155°C):0.025	50	210	145	160	20	7.45	good	good	Invention
6	10	F1(to 200°C)	90	D2(<127°C)	0.200	A4(216°C):0.120	60	C1(147°C):0.080	40	200	135	150	18	7.43	good	good	Invention
7	20	F2(200 to 230°C)	80	D3(<147°C)	0.400	A3(230°C):0.100	25	C1(147°C):0.100 A1(148 to 155°C):0.200	75	210	145	151	11	7.40	good	good	Invention
8	20	G1(>200°C)	80	C1(147°C)	0.150	A3(230°C):0.100	67	A2(127°C):0.025 A1(148 to 155°C):0.025	33	200	140	155	11	7.40	good	good	Invention
9	20	G2(>200°C)	80	A2(127°C)	0.050	A3(230°C):0.020	40	A2(127°C):0.015 C1(147°C):0.015	60	200	135	150	14	7.43	good	good	Invention
10	25	G3(>200°C)	75	A1(148 to 155°C)	0.200	A3(230°C):0.100	50	A1(148 to 155°C):0.025 A2(127°C):0.050 C1(147°C):0.025	50	210	150	160	11	7.42	good	good	Invention

*) content ratio to total amount lubricant
**) total content of lubricant in powdered iron-based mixture
***) content in powdered iron-based mixture
Note: Cross-refer to Table 2 as to the lubricant symbols.

Table 1-2

Green compact Nos.	Die lubricants: Lubricant of higher melting point than compacting temperature: Content* mass%	Die lubricants: Lubricant of higher melting point than compacting temperature: Kind (melting point)	Die lubricants: Lubricant of lower melting point than compacting temperature: Content* mass%	Die lubricants: Lubricant of lower melting point than compacting temperature: Kind (melting point)	Lubricants in powdered iron-based mixtures: Lubricant content** mass%	Lubricants in powdered iron-based mixtures: Lubricant of higher melting point than compacting temperature: Kind (melting point): Content by mass%***	Lubricants in powdered iron-based mixtures: Lubricant of higher melting point than compacting temperature: Content* mass%	Lubricants in powdered iron-based mixtures: Lubricant of lower melting point than compacting temperature: Kind (melting point): Content by mass%***	Lubricants in powdered iron-based mixtures: Lubricant of lower melting point than compacting temperature: Content* mass%	Compacting conditions: Die preheating temperature °C	Compacting conditions: Heating temperature for powdered iron-based mixture °C	Compacting conditions: Compacting temperature °C	Green compacts: Ejection force MPa	Green compacts: Density Mg/m³	Green compacts: Appearance	Green compacts: Sectional structure	Remarks
11	25	H1(>200°C)	75	B1(146°C)	0.350	A4(216°C):0.100	29	A1(148 to 155°C):0.150 C1(147°C):0.100	71	200	145	150	12	7.41	good	good	Invention
12	25	H2(>200°C)	75	C1(147°C)	0.150	A4(216°C):0.100	67	A1(148 to 155°C):0.025 A2(127°C):0.025	33	210	135	140	14	7.42	good	good	Invention
13	30	A3(230°C)	70	A2(127°C)	0.400	A4(216°C):0.100	25	C2(100°C):0.200 A2(127°C):0.100	75	180	115	130	12	7.40	good	good	Invention
14	30	A3(230°C)	70	A2(127°C)	0.050	A4(216°C):0.010 A1(150°C):0.005 C1(147°C):0.015	70	A2(127°C):0.015	30	185	125	135	15	7.45	good	good	Invention
15	30	A3(230°C)	70	C2(100°C)	0.400	A3(230°C):0.100 A4(216°C):0.100 A1(148 to 155°C):0.050 C1(147°C):0.050	75	A2(127°C):0.100	25	185	120	135	13	7.42	good	good	Invention
16	35	A3(230°C)	65	A2(127°C)	0.400	A3(230°C):0.100 A4(216°C):0.100	50	A2(127°C):0.100 C2(100°C):0.100	50	190	130	140	13	7.40	good	good	Invention
17	35	A4(216°C)	65	D2(<127°C)	0.050	A3(230°C):0.025	50	A2(127°C):0.025	50	190	130	140	18	7.43	good	good	Invention
18	35	A4(216°C)	65	A1(150°C)35 D3(<147°C)30	0.200	A3(230°C):0.150	75	A1(148 to 155°C):0.050	25	205	140	155	13	7.40	good	good	Invention
19	40	A4(216°C)	60	B1(146°C)	0.350	A3(230°C):0.200	57	C1(147°C):0.150	43	200	135	150	17	7.42	good	good	Invention
20	40	A4(216°C)20 B1(240°C)20	60	C1(147°C)	0.150	A4(216°C):0.040	27	A2(127°C):0.110	73	200	135	150	17	7.42	good	good	Invention

*) content ratio to total amount lubricant
**) total content of lubricant in powdered iron-based mixture
***) content in powdered iron-based mixture

Table 1-3

Green compact Nos.	Die lubricants: Lubricant of higher melting point than compacting temperature		Die lubricants: Lubricant of lower melting point than compacting temperature		Lubricants in powdered iron-based mixture: Lubricant content** mass%	Lubricants in powdered iron-based mixture: Lubricant of higher melting point than compacting temperature		Lubricants in powdered iron-based mixture: Lubricant of lower melting point than compacting temperature		Compacting conditions: Die preheating temperature	Compacting conditions: Heating temperature for powdered iron-based mixture	Compacting conditions: Compacting temperature	Green compacts: Ejection force	Green compacts: Density	Green compacts: Appearance	Green compacts: Sectional structure	Remarks
	Content* mass%	Kind (melting point)	Content* mass%	Kind (melting point)		Kind (melting point): Content by mass%***	Content* mass%	Kind (melting point): Content by mass%***	Content* mass%	°C	°C	°C	MPa	Mg/m³			
21	40	A4(216°C)20 E2(260°C)20	60	D1(<146°C)	0.050	A4(216°C):0.025	50	A1(148 to 155°C):0.025	50	205	140	155	16	7.42	good	good	Invention
22	40	A4(216°C)20 E3(246°C)20	60	D2(<127°C)	0.200	A4(216°C):0.120	60	C1(147°C):0.080	40	200	135	150	16	7.42	good	good	Invention
23	40	F1(300°C)20 A4(216°C)20	60	D3(<146°C)	0.350	A3(230°C):0.150	43	A1(148 to 155°C):0.100 C1(147°C):0.100	57	205	145	155	14	7.40	good	good	Invention
24	45	E2(260°C)20 G1(>200°C)	55	A1(148 to 155°C)	0.150	A3(230°C):0.100	67	A1(148 to 155°C):0.025 A2(127°C):0.025	33	210	150	160	17	7.44	good	good	Invention
25	50	E2(260°C)25 H1(>200°C)	50	A2(127°C)	0.050	A3(230°C):0.020	40	C1(147°C):0.015 A2(127°C):0.015	60	210	150	160	16	7.43	good	good	Invention
26	50	A3(230°C)25 E2(260°C)25	50	B1(146°C)	0.200	A3(230°C):0.100	50	A1(148 to 155°C):0.025 A2(127°C):0.050 C1(147°C):0.025	50	215	155	165	19	7.42	good	good	Invention
27	50	A4(216°C)20 E2(260°C)20 F1(300°C)10	50	D1(<146°C)	0.350	A4(216°C):0.100	29	A1(148 to 155°C):0.150 C1(147°C):0.100	71	215	155	165	20	7.41	good	good	Invention
28	2	E2(260°C)	98	D2(<127°C)	0.100	A4(216°C):0.050	50	A1(148°C to 155°C):0.025 A2(127°C):0.025	50	220	160	170	19	7.46	good	good	Invention
29	2	E2(260°C)	98	D3(<146°C)	0.400	A4(216°C):0.100	25	C1(147°C):0.200 A2(127°C):0.100	75	220	160	170	17	7.39	good	good	Invention
30	10	A3(230°C)	90	A1(148 to 155°C)	-	-	-	-	-	210	145	160	35	7.31	good	good	Comparative Example

*) content ratio to total amount lubricant
**) total content of lubricant in powdered iron-based mixture
***) content in powdered iron-based mixture

Table 1-4

Green compact Nos.	Die lubricants				Lubricants in powdered iron-based mixture					Compacting conditions			Green compacts				Remarks
	Lubricant of higher melting point than compacting temperature		Lubricant of lower melting point than compacting temperature		Lubricant content** mass%	Lubricant of higher melting point than compacting temperature		Lubricant of lower melting point than compacting temperature		Die preheating temperature	Heating temperature for powdered iron-based mixture	Compacting temperature	Ejection force	Density	Appearance	Sectional structure	
	Content* mass%	Kind (melting point)	Content* mass%	Kind (melting point)		Kind (melting point): Content by mass%***	Content* mass%	Kind (melting point): Content by mass%***	Content* mass%	°C	°C	°C	MPa	Mg/m³			
31	10	A3(230°C)	90	B1(146°C)	0.600	A3(230°C):0.300	50	A2(127°C):0.300	50	190	125	140	29	7.33	marred	good	Comparative Example
32	2	A3(230°C)	98	C1(147°C)	0.150	A3(230°C):0.143	95	C1(147°C):0.0075	5	190	125	140	31	7.34	marred	good	Comparative Example
33	75	A3(230°C)	25	A2(127°C)	0.150	A4(216°C):0.030	20	C2(100°C):0.120	80	180	115	130	17	7.40	good	void	Comparative Example
34	0	-	100	A1(148 to 155°C)	0.350	A3(230°C):0.280	80	A1(148 to 155°C):0.070	20	210	150	160	25	7.42	marred	good	Comparative Example
35	100	A3(213°C)90 A1(148 to 155°C)	0	-	0.200	A3(230°C):0.100	50	A2(127°C):0.100	50	190	125	140	30	7.37	good	good	Comparative Example
36	100	A3(230°C)25 A1(148 to 155°C)	0	-	0.300	A3(230°C):0.225	75	A1(148 to 155°C):0.075	25	100	50	60	25	7.27	good	good	Comparative Example
37	0	-	100	A1(148 to 155°C) A4(216°C)25	0.300	A3(230°C):0.225	75	A1(148 to 155°C):0.075	25	270	210	220	29	7.43	oxidized	good	Comparative Example
38	-	-	-	-	0.600	A3(230°C):0.420	70	A1(148 to 155°C):0.180	30	220	160	170	38	7.35	marred	good	Conventional Example

*) content ratio to total amount lubricant
**) total content of lubricant in powdered iron-based mixture
***) content in powdered iron-based mixture

Table 2

Symbols	Kinds of lubricants		Symbols	Kinds of lubricants	
A1	Calcium stearate	Metallic soap	E1	Polystyrene	Thermoplastic resin
A2	Zinc stearate		E2	Polyamide (nylon 66)	
A3	Lithium stearate		E3	Polytetrafluoroethylene	
A4	Lithium hydroxystearate		F1	Polystyrene elastomer	Thermoplastic elastomer
B1	Straight-chain low-density polyethylene		F2	Polyamide elastomer	
C1	Ethylene bis-stearamide	Amide wax	G1	Graphite	Inorganic lamellar lubricant
C2	Monoamide stearate		G2	MoS_2	
D1	Ethylene bis-stearoamide-polyethylene eutectic	Eutectic mixture	G3	Carbon fluoride	
D2	Ethylene bis-stearoamide-zinc stearate eutectic		H1	Melamine-cyanuric acid adduct (MCA)	Organic lamellar lubricant
D3	Ethylene bis-stearamide-calcium stearate eutectic		E2	N-alkyl aspartate-β-alkyl ester	

Table 3

Sintered Compact Nos.	Die lubricants				Kind of iron-based powder	Graphite content in iron-based powder mixture mass%	Lubricants in powdered iron-based mixtures					Compacting conditions			Green compacts	Sintered Compacts		Remarks
	Lubricant of higher melting point than compacting temperature		Lubricant of lower melting point than compacting temperature				Lubricant content** mass%	Lubricant of higher melting point than compacting temperature		Lubricant of lower melting point than compacting temperature		Die preheating temperature	Heating temperature for powdered iron-based mixture	Compacting temperature	Density	Density	Tensile strength	
	Content* mass%	Kind (melting point)	Content* mass%	Kind (melting point)				Kind (melting point): Content by mass%***	Content* mass%	Kind (melting point): Content by mass%***	Content* mass%	°C	°C	°C	Mg/m³	Mg/m³	MPa	
2-1	75	A3(230°C)	25	A1(148 to 155°C)	a	0.6	0.20	A3(230°C):0.15	75	A1(148 to 155°C):0.05	25	210	150	160	7.42	7.40	830	Invention
2-2	-	-	-	-	a	0.6	0.80	A3(230°C):0.60	75	A1(148 to 155°C):0.20	25	210	150	160	7.32	7.31	740	Conventional Example
2-3	75	A3(230°C)	25	A1(148 to 155°C)	b	0.6	0.20	A3(230°C):0.15	75	A1(148 to 155°C):0.05	25	210	150	160	7.42	7.43	710	Invention
2-4	-	-	-	-	b	0.6	0.80	A3(230°C):0.60	75	A1(148 to 155°C):0.20	25	210	150	160	7.37	7.34	640	Conventional Example
2-5	75	A3(230°C)	25	A2(127°C)	c	0.9	0.20	A1(148 to 155°C):0.09 C1(147°C):0.05	50	A2(127°C):0.01	50	185	120	135	7.23	7.22	810	Invention
2-6	-	-	-	-	c	0.9	0.80	A1(148 to 155°C):0.20 C1(147°C):0.20	50	A2(127°C):0.40	50	185	120	135	7.13	7.12	720	Conventional Example
2-7	75	A3(230°C)	25	A2(127°C)	d	0.9	0.20	A1(148 to 155°C):0.05 C1(147°C):0.05	50	A2(127°C):0.10	50	185	120	135	7.33	7.32	850	Invention
2-8	-	-	-	-	d	0.9	0.80	A1(148 to 155°C):0.20 C1(147°C):0.20	50	A2(127°C):0.40	50	185	120	135	7.25	7.23	760	Conventional Example
2-9	75	A3(230°C)	25	C2(100°C)	e	0.8	0.20	A3(230°C):0.10	50	A2(127°C):0.05 C2(100°C):0.05	50	170	115	130	7.36	7.33	620	Invention
2-10	-	-	-	-	e	0.8	0.80	A3(230°C):0.60	75	A2(127°C):0.10 C2(100°C):0.10	25	170	115	130	7.27	7.14	530	Conventional Example
2-11	75	A3(230°C)	25	C2(100°C)	f	0.8	0.20	A3(230°C):0.10	50	A2(127°C):0.05 C2(100°C):0.05	50	170	115	130	7.25	7.14	680	Invention
2-12	-	-	-	-	f	0.8	0.80	A3(230°C):0.60	75	A2(127°C):0.10 C2(100°C):0.10	25	170	115	130	7.16	7.05	590	Conventional Example
2-13	75	A3(230°C)	25	A1(148 to 155°C)	a	0.8	0.20	A3(230°C):0.15	75	A1(148 to 155°C):0.05	25	210	150	160	7.40	7.39	820	Invention

Sintered Compact Nos.	Die lubricants: Lubricant of higher melting point than compacting temperature: Content* mass%	Die lubricants: Lubricant of higher melting point than compacting temperature: Kind (melting point)	Die lubricants: Lubricant of lower melting point than compacting temperature: Content* mass%	Die lubricants: Lubricant of lower melting point than compacting temperature: Kind (melting point)	Kind of iron-based powder	Graphite content in iron-based powder mixture mass%	Lubricants in powdered iron-based mixture: Lubricant content** mass%	Lubricants in powdered iron-based mixture: Lubricant of higher melting point than compacting temperature: Kind (melting point): Content by mass%***	Lubricants in powdered iron-based mixture: Lubricant of higher melting point than compacting temperature: Content* mass%	Lubricants in powdered iron-based mixture: Lubricant of lower melting point than compacting temperature: Kind (melting point): Content by mass%***	Lubricants in powdered iron-based mixture: Lubricant of lower melting point than compacting temperature: Content* mass%	Compacting conditions: Die preheating temperature ℃	Compacting conditions: Heating temperature for powdered iron-based mixture ℃	Compacting conditions: Compacting temperature ℃	Green compacts: Density Mg/m³	Sintered Compacts: Density Mg/m³	Sintered Compacts: Tensile strength MPa	Remarks
2-14	75	A3(230℃)	25	A1(148 to 155℃)	a	1.1	0.20	A3(230℃):0.15	75	A1(148 to 155℃):0.05	25	210	150	160	7.36	7.36	750	Invention
2-15	75	A3(230℃)	25	A1(148 to 155℃)	a	1.3	0.20	A3(230℃):0.15	75	A1(148 to 155℃):0.05	25	210	150	160	7.32	7.31	580	Invention

*) content ratio to total amount lubricant
**) total content of lubricant in powdered iron-based mixture
***) content in powdered iron-based mixture
Note: Cross-refer to Table 2 as to the lubricant symbols.

Example 3

[0075] A partially alloyed steel powder of a Fe-4Ni-0.5Mo-1.5Cu composition derived by diffusively bonding Ni, Mo and Cu to a pure atomized iron powder was used as an iron-based powder. Iron-based powder mixtures were prepared by mixing this alloyed steel powder with 0.2% by mass of a graphite powder and various lubricants shown in Table 3. The mixing was effected with heat and by use of a high-speed mixer.

[0076] First, a pressure compaction die was preheated at each of the temperatures listed in Table 4. A die lubricant for warm compaction with die electrically charged by a die lubricating system (manufactured by Gasbarre Co.) was jetted into the die and applied to the die surface by means of electrical charging. The die lubricant for warm compaction with die lubrication was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants shown in Table 2, and then by formulating both lubricants as shown in Table 4. The temperature measured on the die surface was taken as a pressure compaction temperature.

[0077] Subsequently, the mold thus treated was filled with a heated iron-based powder mixture, followed by pressure compaction, whereby a rectangular green compact with a size of 10 x 10 × 55 mm was produced. The pressure loading was 686 MPa, and other pressure compaction conditions were as listed in Table 4. A powder compaction lubricant contained in the iron-based powder mixture was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants listed in Table 2, and then by formulating both lubricants as shown in Table 4.

[0078] As a conventional example, a similar rectangular green compact (Green compact No. 38) was formed in the same manner as in Example 4 except that a die was not coated with a die lubricant for warm compaction with die.

[0079] After completion of the compaction, the ejection force was measured.

[0080] With regard to each of the resultant green compacts, the density was determined by Archimedes' principle. Visual inspection was then made of the appearance of the green compact to find faults such as marring, breakage and the like. The green compact was centrally cut, embedded in resin and then abraded, followed by examination of voiding in section on a light microscope.

[0081] The drawing force, density, appearance and sectional structure of the green compact are tabulated in Table 4.

[0082] All the green compacts according to this invention show as low a drawing force after compaction as 20 MPa or below and as high a density as 7.43 Mg/m^3 or above. In addition, each such product causes neither surface oxidation resulting from heating nor faults such as marring, breakage and the like. The sectional structure is normal with the absence of coarse voids.

[0083] The comparative and conventional examples that depart from the scope of the invention suffered a high drawing force exceeding 20 MPa, a low density of less than 7.39 Mg/m^3, or coarse voids near to the sectional surface of the green compact.

[0084] The present invention is highly advantageous in that a high-density green compact is obtainable with superior appearance and sectional structure as well as low drawing force.

Table 4-1

Green compact Nos.	Die lubricants				Lubricants in powdered iron-based mixtures					Compacting conditions			Green compacts				Remarks
	Lubricant of higher melting point than compacting temperature		Lubricant of lower melting point than compacting temperature		Lubricant content** mass%	Lubricant of higher melting point than compacting temperature		Lubricant of lower melting point than compacting temperature		Die preheating temperature	Heating temperature for powdered iron-based mixture	Compacting temperature	Ejection force	Density	Appearance	Sectional structure	
	Content* mass%	Kind (melting point)	Content* mass%	Kind (melting point)		Kind (melting point): Content by mass%***	Content* mass%	Kind (melting point): Content by mass%***	Content* mass%	°C	°C	°C	MPa	Mg/m³	-		
1	2	A3(230°C)	98	A1(148 to 155°C)	0.050	A3(230°C):0.025	50	A1(148 to 155°C):0.025	50	190	150	160	17	7.44	good	good	Invention
2	5	A4(216°C)	95	A2(127°C)	0.150	A3(230°C):0.100	67	A1(148 to 155°C):0.050	33	190	150	160	18	7.43	good	good	Invention
3	5	E1(240°C)	95	B1(146°C)	0.350	A3(230°C):0.200	57	C1(147°C):0.150	43	180	140	150	17	7.44	good	good	Invention
4	10	E2(260°C)	90	A2(127°C)	0.150	A4(216°C):0.050	33	A2(127°C):0.100	67	160	120	130	20	7.43	good	good	Invention
5	10	E3(346°C)	90	D1(<147°C)	0.050	A4(216°C):0.025	50	A1(148 to 155°C):0.025	50	190	145	160	20	7.48	good	good	Invention
6	15	F1(300°C)	85	D2(127°C)	0.200	A4(216°C):0.120	60	C1(147°C):0.080	40	180	135	150	18	7.47	good	good	Invention
7	20	F2(200 to 230°C)	80	D1(<147°C)	0.400	A3(230°C):0.100	25	C1(147°C):0.150 A1(148 to 155°C):0.150	75	190	145	155	11	7.45	good	good	Invention
8	10	G1(>200°C)	90	C1(147°C)	0.150	A3(230°C):0.100	67	A2(127°C):0.025 A1(148 to 155°C):0.025	33	185	140	155	12	7.44	good	good	Invention
9	20	G2(>200°C)	80	A2(127°C)	0.200	A3(230°C):0.080	40	A2(127°C):0.060 C1(147°C):0.060	60	180	135	150	14	7.47	good	good	Invention
10	20	G3(>200°C)	80	A1(148 to 155°C)	0.200	A3(230°C):0.100	50	A1(148 to 155°C):0.025 A2(127°C):0.050 C1(147°C):0.025	50	190	150	160	12	7.45	good	good	Invention

*) content ratio to total amount lubricant
**) total content of lubricant in powdered iron-based mixture
***) content in powdered iron-based mixture
Note: Cross-refer to Table 2 as to the lubricant symbols.

Table 4-2

Green compact Nos.	Die lubricants: Lubricant of higher melting point than compacting temperature – Content* mass%	Die lubricants: Lubricant of higher melting point than compacting temperature – Kind (melting point)	Die lubricants: Lubricant of lower melting point than compacting temperature – Content* mass%	Die lubricants: Lubricant of lower melting point than compacting temperature – Kind (melting point)	Lubricant content** mass%	Lubricants in powdered iron-based mixture: Lubricant of higher melting point than compacting temperature – Kind (melting point): Content by mass%***	Lubricants in powdered iron-based mixture: Lubricant of higher melting point than compacting temperature – Content* mass%	Lubricants in powdered iron-based mixture: Lubricant of lower melting point than compacting temperature – Kind (melting point): Content by mass%***	Lubricants in powdered iron-based mixture: Lubricant of lower melting point than compacting temperature – Content* mass%	Compacting conditions: Die preheating temperature °C	Compacting conditions: Heating temperature for powdered iron-based mixture °C	Compacting conditions: Compacting temperature °C	Green compacts: Ejection force MPa	Green compacts: Density Mg/m³	Green compacts: Appearance	Green compacts: Sectional structure	Remarks
11	25	H1(>200°C)	75	B1(146°C)	0.200	A4(216°C):0.100	50	A1(148 to 155°C):0.060 C1(147°C):0.040	50	190	143	158	14	7.45	good	good	Invention
12	50	H2(>200°C)	50	C1(147°C)	0.150	A4(216°C):0.100	67	A1(148 to 155°C):0.025 A2(127°C):0.025	33	190	155	160	13	7.46	good	good	Invention
13	30	A3(230°C)	70	A2(127°C)	0.400	A4(216°C):0.100	25	C2(100°C):0.200 A2(127°C):0.100	75	160	120	130	12	7.45	good	good	Invention
14	60	A3(230°C)	40	A2(127°C)	0.150	A4(216°C):0.030 A1(150°):0.015 C1(147°C):0.045	67	A2(127°C):0.045	33	165	125	135	14	7.49	good	good	Invention
15	30	A3(230°C)	70	C2(100°C)	0.400	A3(230°C):0.100 A4(216°C):0.100 A1(148 to 155°C):0.050 C1(147°C):0.050	75	A2(127°C):0.100	25	160	120	130	13	7.46	good	good	Invention
16	35	A3(230°C)	65	A2(127°C)	0.200	A3(230°C):0.050 A4(216°C):0.050	50	A2(127°C):0.050 C2(100°C):0.050	50	170	130	140	15	7.45	good	good	Invention
17	35	A4(216°C)	65	D2(<127°C)	0.150	A3(230°C):0.125	83	A2(127°C):0.025	17	170	130	140	18	7.47	good	good	Invention
18	35	A4(216°C)	65	A1(150°C)35 D3(<147°C)30	0.200	A3(230°C):0.150	75	A1(148 to 155°C):0.050	25	190	140	155	14	7.44	good	good	Invention
19	60	A4(216°C)	40	B1(146°C)	0.350	A3(230°C):0.200	57	C1(147°C):0.150	43	180	135	150	17	7.46	good	good	Invention
20	40	A4(216°C)20 B1(240°C)2	60	C1(147°C)	0.150	A4(216°C):0.040	27	A2(127°C):0.110	73	180	135	150	17	7.46	good	good	Invention

*) content ratio to total amount lubricant
**) total content of lubricant in powdered iron-based mixture
***) content in powdered iron-based mixture

Table 4-3

Green Compact Nos.	Die lubricant: Lubricant of higher melting point than compacting temperature: Content* mass%	Die lubricant: Lubricant of higher melting point than compacting temperature: Kind (melting point)	Die lubricant: Lubricant of lower melting point than compacting temperature: Content* mass%	Die lubricant: Lubricant of lower melting point than compacting temperature: Kind (melting point)	Lubricants in powdered iron-based mixtures: Lubricant content** mass%	Lubricants in powdered iron-based mixtures: Lubricant of higher melting point than compacting temperature: Kind (melting point): Content by mass%***	Lubricants in powdered iron-based mixtures: Lubricant of higher melting point than compacting temperature: Content* mass%	Lubricants in powdered iron-based mixtures: Lubricant of lower melting point than compacting temperature: Kind (melting point): Content by mass%***	Lubricants in powdered iron-based mixtures: Lubricant of lower melting point than compacting temperature: Content* mass%	Compacting conditions: Die preheating temperature °C	Compacting conditions: Heating temperature for powdered iron-based mixture °C	Compacting conditions: Compacting temperature °C	Green compacts: Ejection force MPa	Green compacts: Density Mg/m³	Green compacts: Appearance	Green compacts: Sectional structure	Remarks
21	40	A4(216°C)20 E2(260°C)20	60	D1(<146°C)	0.150	A4(216°C):0.100	67	A1(148 to 155°C):0.050	33	190	140	155	15	7.46	good	good	Invention
22	40	A4(216°C)20 E1(240°C)20	60	D2(<127°C)	0.200	A4(216°C):0.080	40	C1(147°C):0.120	60	180	135	150	16	7.45	good	good	Invention
23	50	F1(300°C)25 A4(216°C)25	50	D1(<146°C)	0.350	A3(230°C):0.150	43	A1(148 to 155°C):0.100 C1(147°C):0.100	57	190	145	155	14	7.44	good	good	Invention
24	50	E2(260°C)25 G1(>200°C)25	50	A1(148 to 155°C)	0.150	A3(230°C):0.100	67	A1(148 to 155°C):0.025 A2(127°C):0.025	33	190	150	160	17	7.48	good	good	Invention
25	60	E2(260°C)30 H1(>200°C)30	40	A2(127°C)	0.050	A3(230°C):0.020	40	C1(147°C):0.015 A2(127°C):0.015	60	180	140	150	16	7.47	good	good	Invention
26	70	A3(230°C)35 E2(260°C)35	30	B1(146°C)	0.200	A3(230°C):0.100	50	A1(148 to 155°C):0.025 A2(127°C):0.050 C1(147°C):0.025	50	190	155	165	18	7.46	good	good	Invention
27	80	A4(216°C)30 E2(260°C)30 F1(300°C)20	20	D1<146°C)	0.350	A4(216°C):0.100	29	A1(148 to 155°C):0.150 C1(147°C):0.100	71	190	155	165	19	7.45	good	good	Invention
28	2	E2(260°C)	98	D2(<127°C)	0.200	A4(216°C):0.100	50	A1(148 to 155°C):0.050 A2(127°C):0.050	50	200	160	170	19	7.49	good	good	Invention
29	2	E2(260°C)	98	D1(<146°C)	0.400	A4(216°C):0.100	25	C1(147°C):0.200 A2(127°C):0.100	75	200	160	170	17	7.45	good	good	Invention
30	10	A3(230°C)	90	A1(148 to 155°C)	-	-	-	-	-	190	145	160	35	7.35	good	good	Comparative Example

*) content ratio to total amount lubricant
**) total content of lubricant in powdered iron-based mixture
***) content in powdered iron-based mixture

Table 4-4

Green Compact Nos.	Die lubricants				Lubricants in powdered iron-based mixture					Compacting conditions			Green compacts				Remarks
	Lubricant of higher melting point than compacting temperature		Lubricant of lower melting point than compacting temperature		Lubricant content** mass%	Lubricant of higher melting point than compacting temperature		Lubricant of lower melting point than compacting temperature		Die preheating temperature	Heating temperature for powdered iron-based mixture	Compacting temperature	Ejection force	Density	Appearance	Sectional structure	
	Content* mass%	Kind (melting point)	Content* mass%	Kind (melting point)		Kind (melting point): Content by mass%***	Content* mass%	Kind (melting point): Content by mass%***	Content* mass%	°C	°C	°C	MPa	Mg/m³			
31	10	A3(230°C)	90	B1(146°C)	0.600	A3(230°C):0.300	50	A2(127°C):0.300	50	180	125	150	29	7.36	marred	good	Comparative Example
32	5	A3(230°C)	95	C1(147°C)	0.150	A3(230°C):0.143	95	C1(147°C):0.0075	5	180	125	150	31	7.38	marred	good	Comparative Example
33	90	A3(230°C)	10	A2(127°C)	0.150	A4(216°C):0.030	20	C2(100°C):0.120	80	160	115	130	21	7.42	good	void	Comparative Example
34	0	-	100	A1(148 to 155°C)	0.300	A3(230°C):0.200	67	A1(148 to 155°C):0.010	33	190	150	160	25	7.44	marred	good	Comparative Example
35	100	A3(213°C)90 A1(148 to 155°C)	0	-	0.200	A3(230°C):0.100	50	A2(127°C):0.100	50	170	125	140	30	7.31	good	good	Comparative Example
36	100	A3(230°C)25 A1(148 to 155°C)	0	-	0.300	A3(230°C):0.225 A1(148 to 155°C):0.075	100	-	0	100	60	70	25	7.31	good	good	Comparative Example
37	0	-	100	A1(148 to 155°C) A4(216°C)25	0.300	A3(230°C):0.225	75	A1(148 to 155°C):0.075	25	250	210	220	30	7.47	oxidized	good	Comparative Example
38	-	-	-	-	0.600	A3(230°C):0.420	70	A1(148 to 155°C):0.180	30	200	160	170	38	7.38	marred	good	Conventional Example

*) content ratio to total amount lubricant
**) total content of lubricant in powdered iron-based mixture
***) content in powdered iron-based mixture

Example 4

[0085] The following two different powders were used as iron-based powders; namely (1) a partially alloyed steel powder a of a Fe-4Ni-0.5Mo-1.5Cu composition derived by diffusively bonding Ni, Mo and Cu to a pure atomized iron powder, and (2) a prealloyed steel powder b of a Fe-3Cr-0.3Mo-0.3V composition derived by prealloying Cr, Mo and V.
[0086] The partially alloyed steel powder a, and prealloyed steel powder b were mixed with graphite in the contents shown in Table 5 and the lubricants shown in Table 5, whereby iron-based powder mixtures were prepared. The mixing was effected with heat and by use of a high-speed mixer. The content of graphite is by a mass ratio relative to the total amount of the iron-based powder mixture.
[0087] First, a die was preheated at each of the temperatures listed in Table 5. A die lubricant for warm compaction with die electrically charged by a die lubricating system (manufactured by Gasbarre Co.) was jetted into the die and applied to the die surface by means of electrical charging. The die lubricant for warm compaction with die lubrication was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants shown in Table 2, and then by formulating both lubricants as shown in Table 5. The temperature measured on the mold surface was taken as a pressure compaction temperature.
[0088] Secondly, the die thus treated was filled with a heated iron-based powder mixture, followed by pressure compaction, whereby a rectangular green compact with a size of 10 x 10 x 55 mm was produced. The pressure loading was 686 MPa, and other pressure compaction conditions were as listed in Table 5.
[0089] A powder compaction lubricant contained in the iron-based powder mixture was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants listed in Table 2, and then by formulating both lubricants a lubricants as shown in Table 5.
[0090] With regard to each iron-based green compact thus obtained, the density was determined by Archimedes' principle as in Example 1.
[0091] Subsequently, the iron-based green compact was sintered in a N_2-10%H_2 atmosphere and at 1,130 C for 20 minutes, whereby an iron-based sintered compact was formed. The density of the resultant sintered compact was determined by Archimedes' principle. The test results are tabulated in Table 5. The examples of the invention provides high densities.
[0092] As stated above, the present invention is significantly advantageous in that a high-density green compact can be produced with superior appearance and sectional structure and by single compaction. Drawing of the product from the associated mold is possible at a low force with a prolonged lifetime of the die. Also notably, a high-density sintered compact is easy to produce.

Table 5

Sintered Compact Nos.	Die lubricants				Kind of iron-based powder	Graphite content in iron-based powder mixture mass%	Lubricants in powdered iron-based mixtures					Compacting conditions			Green compacts	Sintered compacts	Remarks
	Lubricant of higher melting point than compacting temperature		Lubricant of lower melting point than compacting temperature				Lubricant content** mass%	Lubricant of higher melting point than compacting temperature		Lubricant of lower melting point than compacting temperature		Die preheating temperature	Heating temperature for powdered iron-based mixture	Compacting temperature	Density	Density	
	Content* mass%	Kind (melting point)	Content* mass%	Kind (melting point)				Kind (melting point): Content by mass%***	Content* mass%	Kind (melting point): Content by mass%***	Content* mass%	°C	°C	°C	Mg/m³	Mg/m³	
2-1	75	A3(230°C)	25	A1(148 to 155°C)	a	0.15	0.15	A3(230°C):0.1	67	A1(148 to 155°C):0.025 A2(127°C):0.025	33	190	150	160	7.49	7.47	Invention
2-2	75	A3(230°C)	25	A1(148 to 155°C)	a	0.30	0.15	A3(230°C):0.1	67	A1(148 to 155°C):0.025 A2(127°C):0.025	33	190	150	160	7.47	7.45	Invention
2-3	75	A3(230°C)	25	A1(148 to 155°C)	a	0.45	0.15	A3(230°C):0.1	67	A1(148 to 155°C):0.025 A2(127°C):0.025	33	190	150	160	7.45	7.43	Invention
2-4	75	A3(230°C)	25	A1(148 to 155°C)	a	0.55	0.15	A3(230°C):0.1	67	A1(148 to 155°C):0.025 A2(127°C):0.025	33	190	150	160	7.39	7.38	Comparative Example
2-5	75	A3(230°C)	25	A2(127°C)	b	0.15	0.20	A1(148 to 155°C):0.05 C1(147°C):0.05	50	A2(127°C):0.10	50	165	120	135	7.34	7.34	Invention
2-6	75	A3(230°C)	25	A2(127°C)	b	0.30	0.20	A1(148 to 155°C):0.05 C1(147°C):0.05	50	A2(127°C):0.10	50	165	120	135	7.32	7.32	Invention
2-7	75	A3(230°C)	25	A2(127°C)	b	0.45	0.20	A1(148 to 155°C):0.05 C1(147°C):0.05	50	A2(127°C):0.10	50	165	120	135	7.30	7.29	Invention
2-8	75	A3(230°C)	25	A2(127°C)	b	0.55	0.20	A1(148 to 155°C):0.05 C1(147°C):0.05	50	A2(127°C):0.10	50	165	120	135	7.25	7.24	Comparative Example

*) content ratio to total amount lubricant
**) total content of lubricants in powdered iron-based mixture
***) content in powdered iron-based mixture
Note: Cross-refer to Table 2 as to the lubricant symbols.

Claims

1. A die lubricant for warm compaction with die lubrication, comprising a mixture of a lubricant having a higher melting point than a preset compaction temperature, and a lower melting point than the compaction temperature, the die lubricant for warm compaction with die lubrication being applicable to the surface of a preheated die by means of electrical charging when a powdered material is compacted by pressure compaction.

2. A die lubricant for warm compaction with die lubrication, comprising a lubricant having a higher melting point than a preset compaction temperature and in a content from 0.5 to 80% by mass, and a lower melting point than the compaction temperature and as the balance, the die lubricant for warm compaction with die lubrication being applicable to the surface of a preheated die by means of electrical charging when a powdered material is compacted by pressure compaction.

3. The die lubricant for warm compaction with die lubrication according to claim 2, wherein the higher-melting lubricant is at least one selected from the group consisting of metallic soap, thermoplastic resin, thermoplastic elastomer, and an organic or inorganic lubricant having a lamellar crystal structure.

4. The die lubricant for warm compaction with die lubrication according to claim 2, wherein the lower-melting lubricant is at least one selected from the group consisting of metallic soap, amide wax, polyethylene, and an eutectic mixture of at least two members thereof.

5. An iron-based powder mixture for warm compction with die lubrication, comprising an iron-based powder and a powder compaction lubricant, wherein the powder compaction lubricant comprises a lubricant having a lower melting point than a preset compaction temperature and in a content from 10 to 75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance.

6. An iron-based powder mixture for warm compaction with die lubrication, comprising an iron-based powder, a powder compaction lubricant and a graphite powder, wherein the powder compaction lubricant comprises a lubricant having a lower melting point than a preset compaction temperature and in a content from 10 to 75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance, and the content of the graphite powder is less than 0.5% by mass based on the total amount of the iron-based powder mixture.

7. The iron-based powder mixture for warm compaction with die lubrication, wherein the content of the powder compaction lubricant is in the range from 0.05 to 0.40% by mass.

8. A process for the production of a high-density iron-based green compact, comprising the steps of: preheating a die at a selected temperature; applying a die lubricant for warm compaction with die lubrication to a surface of the die by means of electrical charging; filling a heated iron-based powder mixture in the die; and then subjecting the powder mixture to pressure compaction at a preset compaction temperature, wherein the lubricant for warm compaction with die lubrication comprises a lubricant having a higher melting point than the compaction temperature and in a content from 0.5 to 80% by mass, and a lubricant having a lower melting point than the compaction temperature and as the balance; and the iron-based powder mixture comprises an iron-based powder and a powder compaction lubricant, the powder compaction lubricant comprising a lubricant comprising a lubricant having a lower melting point than the compaction temperature and in a content from 10 to 75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance.

9. A process for the production of a high-density iron-based green compact, comprising the steps of: preheating a die at a selected temperature; applying a lubricant for warm compaction with die lubrication to the surface of the die by means of electrical charging; filling a heated iron-based powder mixture into the die; and then subjecting the powder mixture to pressure compaction at a present compaction temperature, wherein the die lubricant for warm compaction with die lubrication comprises a lubricant having a higher melting point than the compaction temperature and in a content from 0.5 to 80% by mass, and a lubricant having a lower melting point than the compaction temperature and as the balance; and the iron-based powder mixture comprises an iron-based powder, a powder compaction lubricant and a graphite powder, the powder compaction lubricant comprising a lubricant comprising a lubricant having a lower melting point than the compaction temperature and in a content from 10 to

75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance; and a graphite powder being added in a content less than 0.5% by mass based on the total amount of the iron-based powder mixture.

10. The process according to one of claims 9 and 10, wherein the higher-melting die lubricant is at least one selected from the group consisting of metallic soap, thermoplastic resin, thermoplastic elastomer, and an organic or inorganic lubricant having a lamellar crystal structure.

11. The process according to one of claims 8 and 9, wherein the lower-melting lubricant is at least one selected from the group consisting of metallic soap, amide wax, polyethylene, and an eutectic mixture of at least two members thereof.

12. The process according to any one of claims 8 to 11, wherein the lubricant for warm compaction lubrication is added in a content from 0.05 to 0.40% by mass.

13. A process for the production of a high-density iron-based sintered compact, comprising the step of sintering the high-density iron-based green compact produced by the process according to any one of claims 8 to 12, thereby forming a sintered compact.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP00/05089

A. CLASSIFICATION OF SUBJECT MATTER
Int.Cl⁷ B22F 1/00, 3/02, 3/035, C22C 33/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
Int.Cl⁷ B22F 1/00, 3/02, 3/035, C22C 33/02

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

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Kokai Jitsuyo Shinan Koho	1971-2000	Jitsuyo Shinan Toroku Koho	1996-2000

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP, 10-280005, A (Kawasaki Steel Corporation), 20 October, 1998 (20.10.98) (Family: none)	1-13
Y	JP, 11-193404, A (Hitachi Powdered Metals co., Ltd.), 21 July, 1999 (21.07.99) (Family: none)	1-13
Y	US, 5744433, A (Storstom et al.), 28 April, 1998 (28.04.98) & JP, 10-501270, A (Hoganasu Akuchiboragetto), 03 February, 1998 (03.02.98)	1-13
Y	JP, 9-71801, A (Kawasaki Steel Corporation), 18 March, 1997 (18.03.97) (Family: none)	1-13
Y	US, 5682591, A (Inculet et al.), 28 October, 1997 (28.10.97) & JP, 8-100203, A	1-13

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:
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